CHAPTER 4

Completeness [1]

4-1. Introduction.

The primary objective of this review is to ensure that the data package contains adequate documentation to perform the PB data review. The deliverables that constitute a PB data package are discussed below. Before performing a technical review of the data package, the package should be examined using a checklist to verify that all the required elements are present.

4-2. Minimum Reporting Requirements.

a. As discussed in more detail below, each data package must include a cover page, a table of contents, a Case Narrative, a **Chain-Of-Custody** form, a summary of the environmental sample results (e.g., method of analysis, date analyzed, and amount analyzed), and a summary of the batch QC results (e.g., method blank and LCS results). A summary of all instrument calibration results (i.e., initial calibrations, initial calibration verifications, and continuing calibration verifications) and copies of the sample preparation, standard preparation, and instrument run log sheets must also be included in the data package as described below.

b. The organization of the data package must be such that chemical data are reported on a per **batch** basis. All calibration, method, and batch QC results must be presented on summary forms using a tabular format. The use of CLP standard forms is not necessary. However, submission of standard instrument output alone is unacceptable to satisfy the reporting requirements for PB data packages. Batch QC samples must be clearly linked to their associated environmental samples. Instrument QC samples must be clearly linked to the associated environmental and batch QC samples.

c. The data package must contain sufficient information to determine how the final sample concentrations were calculated from the calibration curves, or, alternatively, how a calibration standard may be expressed as a final sample concentration. In particular, using the sample and calibration summary forms and the standard and sample preparation logs, it must be possible to express the low calibration standard as a sample concentration.

4-2.1. Cover Letter.

The cover sheet includes the following information:

a. Title of Test Report or "Test Certificate."

[1]The evaluation of "completeness" performed during PB data review should not be confused with the "completeness" evaluation that is performed during data usability assessment. The former relates to the data package while the latter is more global in nature and is performed to determine whether or not there is sufficient data of known and acceptable quality to support project decisions.

b. Name and location of laboratory.

c. Laboratory point of contact with phone and facsimile numbers.

d. Name and location of any subcontractor laboratories.

e. Contract number.

f. Client name and address.

g. Project name and site location (if provided by client).

h. Statement of data authenticity and official signature and title of person authorizing the release of the test report.

i. Amendments to previously released reports shall clearly identify previous reports and state the reason(s) for the report amendments..

4-2.2. Case Narrative.

A Case Narrative must be included in each report. The Case Narrative contains tables summarizing the samples received, providing a correlation between field sample numbers and laboratory sample numbers, and identifying which analytical test methods were performed. When analyses are subcontracted to other laboratories, the Case Narrative must clearly specify which laboratory performed each analysis. Samples that were received but not analyzed must also be identified. Extractions or analyses that are performed out of holding times must be appropriately noted. The Case Narrative must define all data qualifiers or flags used. Deviations of any calibration standards or QC sample results from appropriate acceptance limits must be noted and associated corrective actions taken by the laboratory must be discussed. Any other factors that could affect the sample results (e.g., air bubbles in VOC sample vials, excess headspace in soil VOC containers, the presence of multiple phases, sample temperature and sample pH excursions, container type or volume, etc.) must be noted.

4-2.3. Technical Summary.

Summary forms for each sample include the information specified below. Information need not be repeated if noted elsewhere in the data package.

a. Laboratory name and location (city and state).

b. Project name and unique ID number (if specified by client).

c. Field sample ID number as written on custody form.

d. Laboratory sample ID number.

e. Matrix (soil, water, oil, etc.).

f. Sample description.

g. Sample preservation or condition at receipt.

h. Date sample collected.

i. Date sample received.

j. Date sample extracted or prepared.

k. Date sample analyzed.

l. Analysis time when holding time limit is less than 48 hours.

m. Method (and SOP) numbers for all preparation, cleanup, and analysis procedures employed.

n. Preparation, analysis, and other batch numbers.

o. Analyte or parameter.

p. Method reporting limits adjusted for sample-specific factors (e.g., aliquot size, dilution or concentration factors, and moisture content).

q. Method quantitation limits.

r. Method detection limits**.**

s. Final analytical results (e.g., concentrations) *with the correct number of significant figures.*

t. Data qualifiers and definitions of data qualifiers.

u. Concentration units.

w. Dilution factors—All reported data shall reflect any dilutions or concentrations. The dilution factor must be noted on the analytical report. If analyses were performed for both the undiluted and diluted samples, both results must be reported.

x. Percent moisture or percent solids (e.g., soils, sediments, and sludges are typically reported on a dry weight basis).

y. Sample aliquot analyzed.

z. Final extract volume.

4-2.4. Sample Management Records.

These types of records include the documentation accompanying the samples (e.g., original chain-of-custody record, shipping documents, and laboratory notification sheets), records generated by the laboratory which describe the condition of the samples upon receipt at the laboratory (e.g., Sample Cooler Receipt forms, and any records of telephone conversations), and any records generated to document sample custody, transfer, analysis, and disposal.

4-2.5. Batch QC Summary Results.

a. The data package must include all batch QC sample results. This includes method blank (MB), laboratory control sample (LCS), **laboratory control sample duplicate** (LCSD), matrix spike (MS), matrix spike duplicate (MSD), matrix duplicate (MD), and post-digestion spike (PDS) results with the associated acceptance criteria. Summary forms for the MS, MSD, PDS, and LCS, must specify the spiking concentrations, the measured concentrations (e.g., the measured concentrations before and after spike addition for MS and PDS analyses), the **percent recovery,** and the percent recovery acceptance limits for each target analyte. The nature of the recovery acceptance ranges must also be specified (e.g., project-specified acceptance range or in-house laboratory statistical limits). The laboratory's statistical warning and control limits must be specified (in addition to any project required acceptance ranges) for each target analyte for the laboratory control samples (for each preparatory and determinative method).

b. Summary forms for replicate results (e.g., duplicate precision as measured by MS/MSD, LCS/LCSD, or sample/MD pairs), must specify the concentrations of the replicate results, the **relative percent differences (RPDs)** or **percent relative standard deviations (%RSDs)** for each set of replicates (e.g., each duplicate pair), and the acceptance limits (e.g., for the RPDs). The nature of the acceptance limits must also be specified (e.g., project-specified limits or in-house statistical limits for the RPDs).

4-2.6. Standard Preparation Logs.

a. Copies of all relevant *standard preparation log sheets* must be provided for all calibration standards and spiking standards associated with the environmental samples (e.g., the initial calibration, initial calibration verification, continuing calibration verification standards as well as the MS, PDS, and LCS spiking standards). At a minimum, the standard preparation logs must clearly specify the following for all standards:

(1) Source (e.g., manufacturer and lot number for commercial stock solutions).

(2) Composition (e.g., the concentration of all target analytes, surrogates, and internal standards).

(3) Date of preparation and expiration.

(4) Name of the analyst.

(5) ID number of the standard.

(6) Reagents and solvents added to standards (including source and lot numbers).

b. When a standard is prepared via the dilution of a stock solution, the following must be specified: The spiking volume and concentration of the stock solution, and the final volume and concentration of the diluted standard. Copies of manufacturer certificates for commercially purchased stock standards must be included in the standard preparation logs. When the laboratory prepares its own stock solutions, calculations and conversion factors must be shown in the standard preparation log (e.g., using equations or sample calculations).

4-2.7. Sample Preparation Logs.

Copies of the *sample preparation log sheets* must be included in the data package. The sample preparation logs must include the following information:

a. Sample and batch ID numbers.

b. Matrix.

c. Preparatory method (method and laboratory SOP ID number).

d. Date of sample preparation.

e. Initial volume or weight of the sample processed.

f. Final volume of sample processed (e.g., after digestion, extraction or cleanup).

g. Percent moisture (for solid samples).

h. Reagents and solvents added to the samples (including source and lot numbers).

i. Preservation and pH checks or adjustments.

j. Spiking standards (ID number of the LCS, PDS, and MS spiking solutions, volume added, and the final spike concentration).

k. Name of the analyst.

4-2.8. Instrument Run-Sequence Logs.

a. Copies of *instrument analysis log sheets* must be provided for each instrument for each day or analytical shift project samples or associated QC samples were analyzed. *Instrumental analysis logs are particularly important since they provide the basic link between*

the environmental sample analyses and QC data. The run sequence logs must include the following information:

(1) Date of analysis.

(2) Determinative method (including SOP ID number).

(3) Name of the analyst.

(4) Unique ID numbers for environmental and QC samples.

(5) Amount of the sample (instrumentally) analyzed (e.g., the injection volume for chromatographic methods).

(6) Instrument ID number and salient instrument features (column type for chromatographic methods).

(7) Reanalyses and dilution factors (when performed).

b. The run log must clearly identify all QC samples (e.g., continuing calibration verifications). It must also clearly indicate which environmental and batch QC samples are associated with each initial calibration, initial calibration verification (ICV), and continuing calibration verification (CCV). The order in which environmental and QC samples (e.g., ICVs and CCVs) are recorded in the run log must always be consistent with the temporal order in which the samples were actually analyzed. The time of analysis for CCVs and tunings (when performed) must be specified for chromatographic methods. When an autosampler is used (i.e., any device that enables the loading of multiple samples for sequential analysis) the position or sequence number should be recorded in the run log (e.g., the purge port number for purge-and-trap analyses). Lastly, any salient analytical problems (e.g., carry over) must be noted. In particular, *when a QC sample (e.g., a CCV) is reanalyzed, the run log must specify why the reanalysis was performed.*

4-2.9. Traceability.

The data package must clearly demonstrate complete ***traceability*** *of all standards.* For example, unique ID numbers must link all batch QC samples (e.g., MSs and LCSs) in the *instrument run log* to the spiking solutions listed in the *sample preparation log*. The spiking solutions in the *sample preparation log* must be traceable to the original (primary) stock standards via the *standard preparation log*. Similarly, unique ID numbers must link **instrument QC samples** (e.g., CCVs and ICVs) in the *run log* to the corresponding standards in the *standard preparation log*.

4-2.10. Calibration Summary Results.

a. The concentration and corresponding instrumental response (e.g., peak area or peak height) must be reported for each initial calibration standard. When the initial calibration (and quantitation) is performed using internal standards, instrumental response and the corresponding internal standard concentration (or amount and volume of internal standard analyzed) must be

reported for the initial calibration. When more than one internal standard is used, the data package must list the set of target analytes associated with each internal standard. A quantitative "goodness-of-fit" value (e.g., the correlation coefficient, the coefficient of determination, or the **%RSD**) must be reported for the calibration curve of each target analyte and surrogate (when surrogates are used).

b. Plots of the initial calibration curves and instrumental printouts of quantitation summary reports must also be included in the data package for all target analytes. Instrument response must be plotted on the y-axis as the dependent variable and concentration must be plotted on the x-axis as the independent variable. The equation of each calibration curve must also be specified. When calibrations are performed using response factors, the mean response factors must be included with the data package (e.g., in lieu plots and equations for the initial calibrations).

c. The "true" (i.e., reference) concentration (i.e., level spiked), measured concentration, the instrumental response corresponding to the measured concentration, and percent recovery must be reported for each CCV and ICV for each target analyte and surrogate (when surrogates are analyzed). When internal standards are used, the instrumental response for each internal standard and the corresponding internal standard concentration (or amount and volume of internal standard analyzed) must also be reported. When an ICV is not performed, the data package must clearly indicate (e.g., in the standard preparation logs) whether an independent-source standard was used to spike the CCVs or LCSs.

4-2.11. Chromatographic Methods for Organic Target Analytes.

The additional reporting requirements specified in this section of the document apply only to chromatographic methods for organic target analytes.

4-2.11.1. Initial Calibration.

When the initial calibration is performed using response factors, the **response factor** for each initial calibration standard and the mean response factor for each analyte and surrogate must be reported. The %RSD must also be reported for each set of initial calibration standards. A reporting format similar to the CLP "Initial Calibration Summary Form" for the GC/MS BNA and VOA analyses is recommended. Response factors must also be reported for CCVs and the initial calibration when minimum response factors are specified by the analytical method (e.g., GC/MS Methods 8260A and 8270B). (Note that this does not imply that regression analysis cannot be used to perform initial calibrations for these methods.)

4-2.11.2. Internal Standard Summary Information.

When internal standards are used, the internal standard areas and retention times must be summarized for each batch QC and environmental sample in a tabular format (e.g., using summary forms similar to the CLP "Internal Standard Area And RT Summary" forms). The internal standard retention time and retention time windows must also be specified for the most recent associated CCV. The area counts and area count acceptance ranges for the associated mid-level ini-

tial calibration standard must be specified on the internal standard retention time and peak area summary form.

4-2.11.3. Surrogate Results.

The expected concentrations of the surrogates, the surrogate recoveries, and surrogate recovery acceptance ranges must be reported for all the environmental samples, batch QC samples, continuing calibration verifications, and initial calibration verifications. The surrogate recoveries for the environmental and batch QC samples must be summarized in a tabular format (e.g., using forms similar to the CLP "Surrogate Recovery" forms). The nature of the surrogate recovery acceptance ranges must also be specified (e.g., project-specified versus in-house statistical control ranges). The laboratory's statistical warning and control limits must be specified (in addition to any project required acceptance range) for the surrogates in the laboratory control samples or method blanks (for each preparatory and determinative method).

4-2.11.4. Chromatographic Methods With 2-D Detectors.

a. Additional reporting requirements are required for chromatographic methods with 2-D detectors (e.g., FIDs, PIDs, and ECDs). When chromatographic analyses are performed using second column confirmation, the results for both analytical columns must be reported (e.g., surrogate recoveries, sample results, and initial and continuing calibration results).

b. The results for the environmental samples must be reported using summary forms similar to the CLP "Pesticide Identification Summary For Single Component Analytes" and the "Pesticide Identification Summary For Multicomponent Analytes." Retention times and retention time windows must be reported for all single component standards (e.g., initial calibration, continuing calibration verification, and internal standards), target analytes, and surrogates or both the primary and confirmatory columns. For multi component analytes, the retention times and retention time windows should be specified for at least three to five characteristic peaks. (Note that retention time and retention time windows must be reported for both the primary and confirmatory columns.) In addition, the results (e.g., concentrations) from the primary and secondary columns, as well as the corresponding RPDs must be reported.

4-3. Evaluation of Completeness.

a. Use professional judgement to determine the degree to which missing information can be tolerated. Distinguish sporadic occurrences of missing noncritical data from systematic noncompliances. For example, if the LCS and method blank results were not included, the data would typically be of unknown quality and would be rejected. However, the entire data package would not typically be rejected if the dilution factor were not specified for one of the environmental samples.

b. If the data package is not substantively complete, (i) missing information must be requested from the laboratory, (ii) the data package must be rejected, or (iii) a limited data review must be performed. When the data package is grossly deficient, the reviewer should consult with the Project Manager to determine which option is most appropriate for the data objectives of the

investigation. For example, if missing deliverables cannot be obtained from the laboratory or if data evaluation cannot be delayed because of scheduling constraints (e.g., because time-critical decisions must be made), a more limited data review may be appropriate. If the data are being used to support critical decisions (e.g., for litigation), it may be necessary to reject the data when standards are not traceable or the COC is missing (since the integrity the data has not been definitively demonstrated).

c. When QC sample results are not included in the data package, it may be possible to use the available results to evaluate or to make inferences concerning the quality of the data. *However, this approach must be used with caution since the missing QC samples may have been analyzed by the laboratory but not reported because of QC failure.* Some evaluation strategies for incomplete data are discussed below.

4-3.1. Missing Blanks.

a. When a blank is missing, a higher **hierarchy** blank may be used to qualify the corresponding field samples for contamination. For example, if the method blank is not available (e.g., because it was not processed with the batch of samples), then field samples may be qualified using another associated blank that was processed with the batch of samples. In particular, if a rinsate blank or trip blank were analyzed with the batch of samples, rather than rejecting the data, the data could be evaluated using the trip blank or rinsate blank data. The trip blank or rinsate blank would be indicative of the accumulative field and laboratory contamination.

> Note: This approach will not be appropriate when the field blank is not processed in the same batch (or in the same manner) as the environmental samples for which it is assumed to represent.

b. Similarly, an environmental sample in the preparation batch for which no target analytes were detected may serve as a *field blank* (since such a sample would demonstrate the lack of systematic field and laboratory contamination).

> Note: Since samples are processed through a variety of handling, preparatory, and analysis procedures, blanks are typically collected during various stages of these procedures in a manner which would establish the source of contamination and enable the implementation of corrective action. In theory, if a **comprehensive blank** is truly representative of all contamination that could have been introduced from the time of sample collection to analysis (especially if the blank is free of contamination), then missing lower **hierarchy** blanks should not affect the data qualification. However, it should be noted that, in practice, it is difficult to obtain a representative **comprehensive blank**. For example, common laboratory contaminants such as methylene chloride can appear in blanks in a sporadic manner (e.g., may be present in a method blank but may not be present in a higher hierarchy blank such as a field blank).

c. When the method blank is missing and a higher hierarchy blank is not available to evaluate the data, qualification may be required for the corresponding field sample results. (Contractual corrective actions may also be required for missing method blanks, because labo-

ratories are usually required to process a method blank with each batch of field samples.) *In general, nondetections must not be qualified (unless other QC problems are present).* Detections must be qualified with the R, X, or N flag. Project-specific DQOs and professional judgment must be used to determine which flag is more appropriate. However, *the R or X flag must be used to qualify detections in the absence of a technically defensible rationale for using the N flag.* Some situations for which the use of the N flag may be appropriate are presented below.

d. It may be appropriate to qualify an analyte detection with the N flag if the analyte was reliably detected in other site field samples that were processed in a separate batch with an acceptable method blank. Since the probability of external contamination as the source of a detection typically varies inversely with the magnitude of the detected concentration, it may be appropriate to qualify very high level detections with the N flag (e.g., detections ten times greater than the MQL). If an action level is available, it may be appropriate to qualify detections less than the action level (especially very low-level detections) with the N flag. However, in the absence of a technically defensible rationale to do otherwise (i.e., when information supporting the validity of detected concentrations is not available), detections greater than an action level must be qualified with the R or X flag when blank results are not available.

4-3.2. Missing Laboratory Control Samples.

In general, when LCS results are not available for a particular batch of field samples, the data are of unknown quality and the associated field sample results must be qualified with the R or X flag. Since a laboratory is typically required to process at least one LCS with each batch of samples, contractual corrective action for unacceptable performance may be appropriate. Possible exceptions are discussed below.

4-3.2.1. Matrix Spike Data.

a. If the LCS is missing but all target analytes are present in the MS and precision information is available from the MS/MSD or MD, then the associated field samples may be qualified (for bias and precision) using the MS/MSD or MS/MD results since this data gives bias and precision information for the overall method. In particular, if all the MS/MSD or MS/MD recoveries and RPDs are in control, then the sample results need not be qualified. When the MS/MSD or MS/MD is not in control, the associated field samples must be qualified but the QC failure may be due to poor laboratory method performance (rather than matrix interference).

b. If the LCS is missing and only a subset of the target analytes is present in a MS/MSD or MS/MD pair, then qualify the associated field samples as discussed above for the subset of spiked analytes. Reject or tentatively reject the associated field sample results for the unspiked target analytes. If the data are being used to support noncritical decisions, (for target analytes not present in the matrix spike), it might be appropriate to reject or tentatively reject nondetections but to qualify detections as estimated.

4-3.2.2. Surrogate Spike Data.

Surrogate recoveries may be used to make inferences about the performance of organic analyses. If acceptable surrogate results are available and the data will be used to support noncritical decisions, it is recommended that the data be qualified as estimated rather than as rejected. *However, this approach must be used with caution since the surrogates may not be representative of all the target analytes* (e.g., although ketones are often target analytes for SW-846 Method 8260B, none of the surrogates recommended in the method are ketones).

4-3.2.3. CCV Data.

When all environmental and QC samples undergo the same preparatory and determinative processes or when significant sample preparation is not performed (e.g., aqueous VOCs by purge-and-trap GC/MS and aqueous anions by ion chromatography), CCVs are essentially LCSs. Method performance may be evaluated using the CCV results. If the CCV results are acceptable, then the data would not be qualified on the basis of the missing LCS results.

4-3.3. Missing Detection Limits.

a. Detection limits must be included in the data package. Laboratory detection limits are typically the **method detection limits (MDLs)** defined in 40 CFR, Part 136, Appendix B. *Instrument detections limits (IDLs) must not be viewed as a substitute for MDLs.* For example, *MDLs* may be significantly higher than *IDLs* when an analytical method involves extensive sample preparatory procedures.

> Note: Laboratories frequently perform an MDL study for a single instrument and erroneously report the resulting MDLs as applicable to all similar instruments used for the same analytical method. Method detection limits are matrix, method, and *instrument-specific*. For example, if a laboratory performs Method 8260B using six GC/MS instruments, then six sets of MDL studies must be available or sensitivity must otherwise be demonstrated for all six instruments. A valid conservative approach may consist of performing a separate MDL study for each instrument but reporting only the highest MDLs for each instrument.

b. When detection limits are not available (i.e., not included in the data package), the laboratory should be contacted for this information or the method reporting limits (MRLs) for nondetections must be set equal to the MQLs (e.g., when the laboratory lists reporting limits that are less than the MQLs). However, the MQLs must be verified as discussed in Chapter 6.

(1) If an action level is unavailable, then qualify all detections less than the MQL with the J flag. Report all nondetections as "< MQL" or "MQL U," where "MQL" denotes the numerical value of the method quantitation limit.

(2) If an action level is available, then compare the MQL to the action level. If the MQL is less than the action level, then qualify detections and nondetections as described above. *How-*

ever, if the MQL is greater than the action level, adequate sensitivity has not been demonstrated. Qualify nondetections with the X or XU flag and qualify detections less than the action level with the X flag. A detection below the action level does not demonstrate that the target analyte is actually present in the environmental sample below the action level because analytical uncertainty is large or undefined below the quantitation limit. Detections greater than the action level but less than the MQL may be qualified with the X flag but, at a minimum, must be qualified with the J flag. When resampling or reanalysis cannot be performed, the use of the J flag normally constitutes the more conservative approach.